

**NANOSTRUCTURES AND ENHANCED PROPERTIES IN TUNGSTEN AND ITS ALLOYS  
PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING.**

**Interim Report No. 0002**

by

**R.Z. Valiev and I.V. Alexandrov  
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<b>13. ABSTRACT</b> The present report presents the results of an experimental investigation and numerical simulation of the process of equal-channel angular pressing with a backpressure equal to $2.4 \sigma_s$ , which have allowed to increase the accumulated strain rate up to $\epsilon=7$ , what allows to obtain ultrafine-grained structure with a grain size less than $0.5 \mu m$ . Note should be made that in that case the punch loading was close to its calculated value, which is in conformity with the strength characteristics of the material and the die-set design. With the purpose of studying the influence of initial state on the ultrafine-grained structure formation there were carried out comparative investigations of the behavior of single- and polycrystalline tungsten during severe plastic deformation process. A possibility of reducing the severe plastic deformation temperature when using special sheathing for tungsten billets is being investigated. There were started investigations of the microstructure refinement of the W-2%Re alloy with enhanced strength and ductility at severe plastic deformation.				
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The results of the investigation carried out in the frame of the first period of the present contract N6871-01-M-5641 showed, that the problem of obtaining bulk billets with ultrafine-grained (UFG) (less than  $1\mu\text{m}$ ) structure by severe plastic deformation (SPD) by means of equal-channel angular (ECA) pressing in low-ductility and hard-to-deform materials, tungsten and its alloys are referred to, is a challenging task, which requires developing of special approaches and treatments, which allow to increase the workability of the above mentioned materials. In this connection, experimental and theoretical investigations were carried out in the frame of the second period. They were aimed at:

- 1) numerical modelling of the flow of material for studying the dynamics of strain accumulation, as well as stress analysis of contact, normal and shear stresses in intersecting channels during ECA pressing with backpressure;
- 2) approving and investigating of ECA pressing in a new special die-set, which allows to achieve enhanced (up to  $2.4\sigma_s$ ) magnitudes of backpressure in order to prevent the destruction of billets;
- 3) studying the influence of initial microstructure on the formation of an ultrafine grain in W as a result of severe plastic deformation, processed by high pressure torsion (HPT) and equal-channel angular pressing;
- 4) studying the refinement of W-Re alloys microstructure during SPD.

It was pointed out in the result of numerical modelling, that normal stresses in the channels of the ECA pressing die-set when the imposed backpressure up to  $2.4\sigma_s$  is applied to, does not exceed 1400 MPa. The given stress level is acceptable from the point of view of strength and preserving the integrity of the die-set. Testing of the new die-set with backpressure demonstrated the possibility of increasing the accumulated strain rate, before first cracks on the samples appeared, up to  $\epsilon=7$  in comparison with earlier achieved rates  $\epsilon=5.6$ . A pivotal role for preserving correct cylindrical shape of the deformed billets after ECA pressing and lessening of oxidation plays the geometry of sheathing, the billet was enclosed in. To solve this problem, on the third stage of the present project will be carried out experimental investigations and the computer modelling of the influence of geometric parameters on sheathing, providing the observance of temperature rate and protection of W-billet from oxidation under high temperatures during ECA pressing. Accordingly, will be created the necessary prerequisites for successful realization of the processes of ECA pressing of not only W-bulk billets, but also of Mo and Ta billets. It will be carried out on the next stages of the given project.

One of the main questions in forming ultrafine-grained structures by SPD is the role of the initial microstructure [1, 4]. In this connection, we conducted comparative investigations of the processes of ultrafine-grained structure formation during SPD of single and polycrystalline W.

Single crystalline W, obtained by double remelting in accordance with the arc-plasmas method and containing 0.2 C, 1.6 O<sub>2</sub>, 24 P, 0.5 Si, 80 Fe, 1500-200 Mo, 1 S, 0.1 Cr, 0.1 Ni,  $4\text{ Ti} \times 10^{-4}$  mass. % of admixtures, was purchased in Baykov's Institute of Metallurgy and Material Science RASc (Moscow). The ingot had a form of ellipsoid with a big axis, which coincide with crystallographic direction  $\langle 111 \rangle$ . The samples for HPT were carved by the spark cutting method in a disc form 10 mm in diameter and 0.5 mm in thickness with a normal to a discs plane, which is parallel to the direction  $\langle 111 \rangle$ . The sample for the ECA pressing had a shape of a cylinder 60 mm in length and 15 mm in diameter of the cross-section. The length of the cylinder coincided with the crystallographic direction  $\langle 111 \rangle$ . HPT of the single crystalline samples was realized under the pressure of 6 GPa with a number of turns equal to 5, at a temperature of 500 °C. As a result discs without visible cracks were obtained. The discs are 10 mm in diameter and 0.3 mm in thickness. On the third stage of the project execution the ECA pressing of the single crystal will be carried out. Also TEM and X-ray investigations of the microstructure and microhardness of these samples previously subjected to HPT and ECA pressing will be conducted.

As it is well-known, both a significant increase in ductility and an increase in strength can be achieved as a result of alloying by Re ("rhenium effect") [3]. According to literature sources [2], in this case not only alloys with the maximum content of Re in a solid solution (25%) reveal increased ductility, but also W-Re alloys with a small content of Re about 2%. In this connection within the frame of the second stage of the present project the W-2%Re alloy developed and manufactured in the Material Science Institute of the Ukraine National Academy of Sciences (Kiev) was purchased. The

alloy was obtained by sintering of billets from dispersed powders and their consequent rolling into a plate of 2 mm in thickness with a strain of 93%. The structure of the material in the as-delivered state represented grains elongated in the direction of rolling with the dimensions of 75  $\mu\text{m}$  and 20  $\mu\text{m}$  in the longitudinal and the cross sections, correspondingly. Transmission electron-microscopy investigations have demonstrated that rolling of the powder alloy W-2%Re results in formation of a developed substructure with elongated subgrains the mean grain size of which equals 1.5  $\mu\text{m}$ .

HPT of the W-2%Re alloy discs was performed at a temperature of 500  $^{\circ}\text{C}$  under pressure of 6 GPa with the number of turns equal to 5. To torsion were subjected samples in the as-delivered state and after annealing for 30 minutes at 1400  $^{\circ}\text{C}$ . As a result of the annealing a recrystallized microstructure with a mean grain size of 25  $\mu\text{m}$  was formed in the samples.

HPT of the previously recrystallized alloy made it possible to obtain samples without visible cracks. At the same time, torsion of the rolled alloy led to development of radial microcracks which was evidently caused by the appearance of higher internal microstresses than in the case of the rolled alloy.

Further research will be oriented towards study of the structure and mechanical properties of the obtained SPD W-Re samples.

Apart from the tasks mentioned above, in the frame of the third stage of the present project problems related to purchase of Mo and Ta and possibility of forming a UFG microstructure by SPD in these materials will be tackled. The investigation of the mechanical properties of W samples after SPD is planned in order to determine the influence of the UFG structure on the temperature of the brittle-ductile transition.

The presentations for the International Conference on Refractory & Hardmetals in London (28.10-30.10, 2002) will be prepared conjointly with Dr. Robert J. Dowding (U.S. Army Research Laboratory) on the basis of the results of the investigations conducted.

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